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# SANDY CRYSTALS IN ICE CREAM: THEIR SEPARATION AND IDENTIFICATION

By HARPER F. ZOLLER, *Chemist*, and OWEN E. WILLIAMS, *Dairy Manufacturing Specialist, Dairy Division, Bureau of Animal Industry, United States Department of Agriculture*[1]

Ever since Sato (*6*)[2] isolated crystals of lactose, sucrose, calcium phosphate, calcium citrate, etc., from the crystalline sediment occurring in sweetened condensed milk, there has been considerable speculation as to which of these substances are responsible for most of the sandiness found therein.[3]

The ice-cream industry utilizes large quantities of condensed milk, particularly evaporated skim milk and sweetened condensed whole milk. The tendency during the last few years toward building up the total solids of the ice-cream mix with milk solids other than milk fat has created a demand for the products mentioned. With their use there arose simultaneously the problem of "sandy" ice cream. Not infrequently wholesale quantities of ice cream develop this sandy or crystalline texture—sometimes large, hard crystals, sometimes fine and gritty, like starch. This wide variation in the size of the crystals caused much perplexity as to their composition in view of the work cited above on condensed milk. Since ordinary ice cream contains from 12 to 16 per cent of sucrose it was but natural that many should believe the sandiness, especially the large crystals, to be due to this substance; the finer might be due to any of the suspected substances.

In order to settle this question of "what constitutes sandiness" we took up the problem of isolating the "sandiness" after it had formed and of identifying it.

## DEVELOPMENT OF SANDY CRYSTALS

A large quantity of a high solids-not-fat ice cream was prepared in a regular commercial power freezer. The composition of the mix was as follows: Fat, 10 per cent; sucrose, 14 per cent; milk solids not fat, 12 per cent. The raw products were ripened 40 per cent cream, evaporated skim milk, and pure cane sugar. The mix was frozen in the regular time with brine at about 10° F. (− 12.22° C.). It was immediately packed, after freezing, in 2-quart tinned cans and placed in the hardening

[1] The authors express their indebtedness to G. L. Keenan and E. T. Wherry, of the Bureau of Chemistry, to the former for his courtesy in preparing the photomicrographs and to the latter for checking the crystallographic fea tures of lactose.

[2] Reference is made by number (italic) to "Literature cited," p. 795.

[3] It would be unfair to create the impression that Sato actually isolated sucrose crystals from all samples of condensed milk. In fact, the sucrose crystals were isolated only rarely from condensed milk, and in those cases he specifically states that they were condensed to an unusually low water content.

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box. This box was insulated with wood and held a hardening mixture of ice and rock salt. The ice cream was repacked each day in this box.

On the fourth day very fine sandy crystals appeared. The crystals were developed by allowing the cans to become "heat shocked" (by setting them in contact with the air of the room for about 20 minutes) every other day after the sandiness first became perceptible.

The dimensions of the crystals were observed frequently through the low-power objective of a compound microscope. When the sandiness first became perceptible to the tongue and between the fingers, the crystals were clearly visible in the microscope. The general form of the crystals remained the same throughout 20 days' observation, although they grew much in size during this interval.

When the crystals had grown to a sufficient size they were separated from the parent mass. Plate 137 illustrates the appearance of the crystals in the frozen ice cream described above, when a drop of the ice cream was placed upon a glass slide and then viewed through the microscope in polarized light. The crystals showed plainly the natural maize-shaped form of lactose hydrate.

## SEPARATION OF THE CRYSTALS

The sandy crystals were separated from several different preparations of ice cream by the following method: Frozen cream was allowed to melt at the temperature of the laboratory, about 25° C. When completely melted it was poured into large centrifugal tubes of 100 cc. capacity and centrifuged at 2,000 revolutions per minute for 10 minutes. The fat and liquid layers were poured from the sediment; the liquid portion was now entirely free from sandiness, the latter having been thrown down in the form of sediment. A drop of the sediment examined on the microscopic slide seemed to be made up entirely of crystals of one form—some large and some small.

Before the crystals were proved to consist entirely of pure lactose they were isolated from the sediment for the purpose of holding any sucrose or lactose crystals in the crystalline form. This was effected by saturating water with all the sucrose and lactose that it could hold at 2° C. It was then warmed to 5°, and the sediment in each tube was thoroughly shaken with about 75 cc. of it. Then the tubes were again centrifuged. This operation was repeated with the addition of 50 per cent acetone to the washing fluid. The product was further freed from liquid on a Büchner funnel.

The crystals were obtained in a pure white condition. More than ½ kilo was obtained in this manner. They were dried to constant weight on filter paper in a boiling-water oven. Plate 138, A, illustrates the appearance of these isolated crystals. They show plainly the effect of the washing mixtures but retain their characteristic maizelike or wedge-like form.

## IDENTIFICATION OF THE CRYSTALS AS LACTOSE HYDRATE

### MICROSCOPICAL EXAMINATION

The ease with which milk sugar usually crystallizes and the very characteristic form of its crystals suggest the use of the microscope as the most accurate means of identifying it. As mentioned in the earlier part of the text it is possible to detect and follow microscopically the growth of the crystals in the ice cream.

For the purpose of identification of the sandy crystals it was necessary to have at hand pure specimens of lactose and sucrose crystals. Rather than accept the market lactose as standard for recrystallization a quantity of pure lactose was obtained by evaporating some milk whey from cheese-making in a vacuum pan to a concentration of 8 parts by weight of whey to 1 part by weight of product. The resulting lactose was filtered and purified according to the customary methods.

The crystalline lactose thus obtained was the $\alpha$ form, or commonly recognized lactose hydrate ($C_{12}H_{22}O_{11}.H_2O$). According to Groth (*1*, *p. 450*) its crystallographic features are—

Monoclinic-sphenoidal. Cleavage in three directions nearly at right angles. Refractive indices, $\alpha = 1.517$; $\beta = 1.542$; $\gamma = 1.550 \pm 0.005$ $Bx^a \wedge c = 10°$, $\wedge a = 99°$. $2E = 33\text{-}1/2°$. Sign $-$, sp. gr. $1.525 - 1.534$.

Some of these crystals were placed upon a microscopic slide, and a photomicrograph was obtained with polarized light. The photograph exhibits the characteristic tomahawk-shaped crystals which are always presented by $\alpha$-lactose when the crystals are allowed to develop spontaneously below 93° C. (*3*). When they crystallize at 93°, or a little above, lenticular needles appear ($\gamma$ or anhydride form) (*2*), which are markedly different to the eye. These gradually transform into the tomahawk type as the temperature drops below 93°. The photomicrograph of these lactose-hydrate crystals is shown in Plate 138, B.

The comparison of these pure lactose crystals with those within the ice cream in Plate 137, as well as with that of the sandy crystals separated in a pure condition from the same ice cream, shown in Plate 138, A, shows the sandy crystals to be identical in form with those of pure lactose. It is but natural that we should realize the effects shown in Plates 137 and 138, A, of substances tending to hinder perfect growth of the crystals. Also the solvent action of water is evident in the partially dissolved facets and less sharp outlines. Otherwise there is no difference in the appearance between the crystals in Plate 138, B, and those in the first three figures.

A fully developed crystal of sucrose is shown in Plate 138, C. The sucrose crystals were grown within a pectin gel from a supersaturated sucrose solution (500 per cent solution) at ordinary temperature. There is not the least resemblance between this crystal and those isolated from ice cream.

## CHEMICAL ANALYSIS

Some of the isolated sandy crystals mentioned in the section on separation, and similar to those photographed in Plate 138, A, were analyzed for lactose, without inversion, according to the Munson and Walker method,[1] described by Leach (*4, p. 598*). The average of three determinations gave 99.86 per cent calculated as lactose hydrate from Munson and Walker's tables. It is evident that the quantity of sucrose appearing as sandy crystals is insignificant.

## DISCUSSION

The results of our experiments upon a number of sandy ice creams lead us to conclude definitely that the sandiness is due to the crystallization of lactose from a supersaturated solution.

The suspicion, fostered by a number of persons in the dairy industry, that sucrose is responsible for this sandiness is not founded on scientific principles. The solubility, recorded by many investigations, of lactose in water at 10° C. is about 17 gm. per 100 gm. of water. The solubility of sucrose at 12° is given as 198.6 gm. per 100 gm. of water. When these solutions are cooled they become supersaturated, provided they contain the above-mentioned quantities of the respective sugars. In sucrose solutions, however, we meet with the experience of the sugar technician, Prinsen Geerligs (*5, p. 301*), who tells us that sucrose solutions frequently act like glue at low temperatures and can be supercooled to a state in which they can not be poured. In the highly supersaturated, supercooled condition it is very difficult to induce them to crystallize. When accomplished it requires weeks and even months for the crystals to develop in size, particularly if much colloidal material, such as gum, pectin, or protein, is present in the solution. It required several months for the perfect crystal of sucrose shown in Plate 138, C, to develop even to this size.

In the case of lactose, because of its low solubility in water, compared to sucrose, it is more difficult to form supersaturated solutions of any great magnitude. When its solutions are concentrated it crystallizes without much excitation. Indeed, it will persist in crystallizing even in the presence of complex protective substances which check crystallization of many other compounds.

An ice-cream mix of 10 per cent fat, 12 per cent milk solids not fat, and 14 per cent sucrose contains, if we assume that the two sugars compete for only the water in the mix, about 22 per cent sucrose solution and about 10.5 per cent lactose solution. When we recall the fact that sucrose is about 10 times as soluble as lactose in water, we readily see that the physical conditions existing in frozen ice cream are unsuited to the crystallization of sucrose.

[1] MUNSON, L. S., and WALKER, Percy H. UNIFICATION OF REDUCING SUGAR METHODS. *In* Jour. Amer. Chem. Soc., v. 28, no. 6, p. 663–686, 1906; v. 29, no 4, p. 541–554, 1907; Final tables *in* U. S. Dept. Agr. Bur. Chem. Bul. 107 (rev.), p. 241–251, 1908; A correction, U. S. Dept. Agr. Bur. Chem. Circ. 82, 6 p., 1911.

SUMMARY

(1) The "sandiness" in ice cream has been isolated and identified as lactose.

(2) The growth of the "sand" crystals can be followed satisfactorily with the microscope.

(3) The form of lactose appearing in the ice cream is the normal $\alpha$ crystal which crystallizes from water solutions in tomahawk-shaped prisms and from protein solutions in more rugged or maize-shaped crystals.

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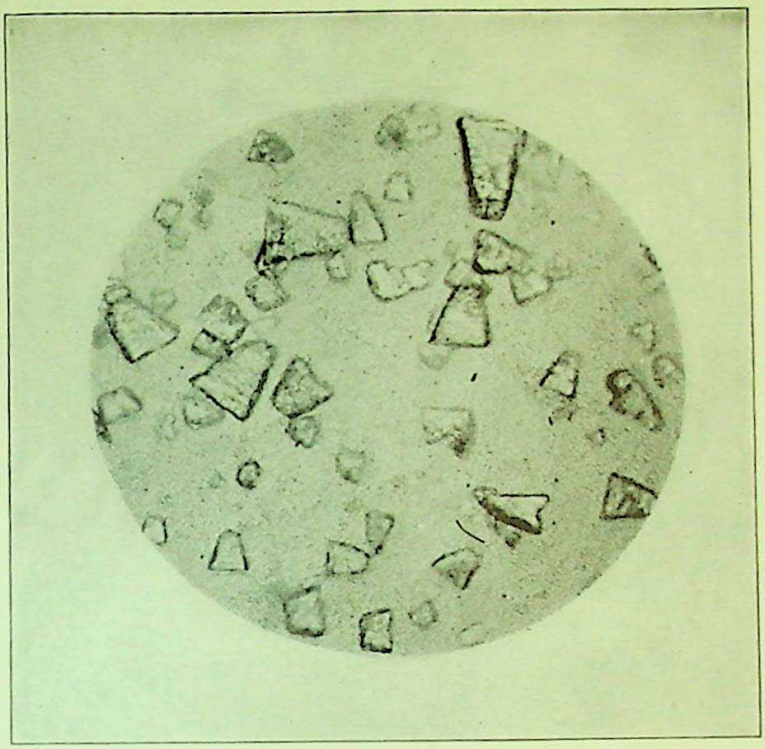
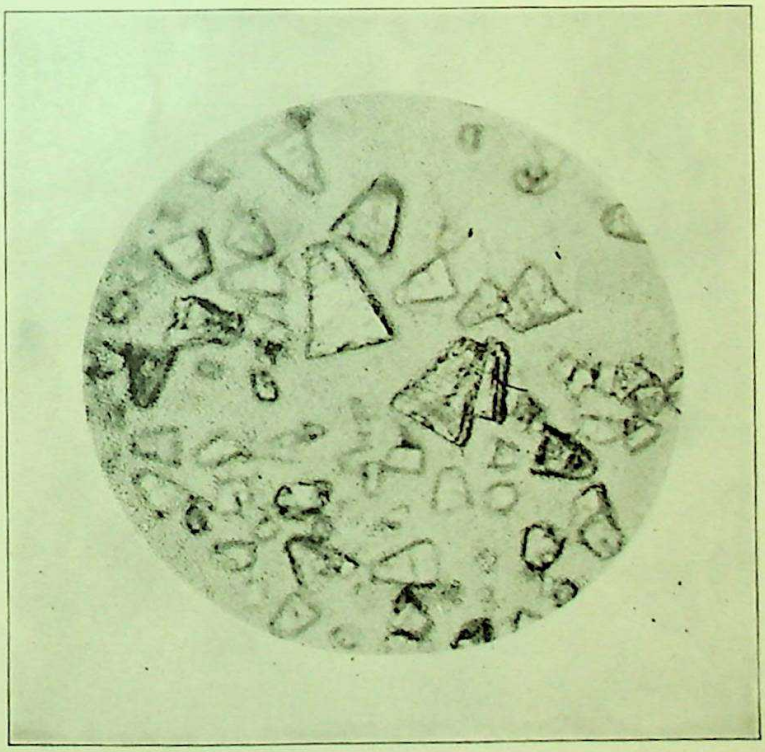
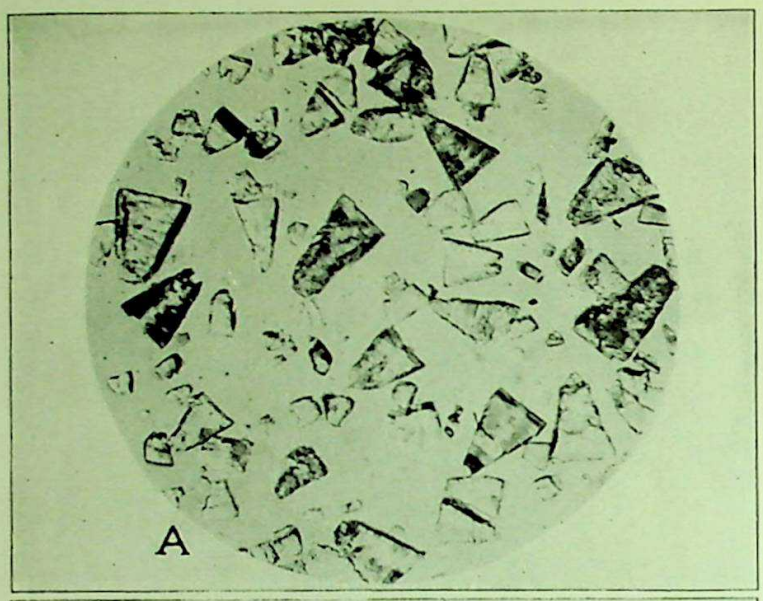
PLATE 137

Photomicrographs of the "sand" crystals in ice cream, made by placing a drop of the melted ice cream upon a microscopic slide and covering it with a cover glass. This was then placed upon the stage of the photographic microscope and photographed with polarized light. × 90.



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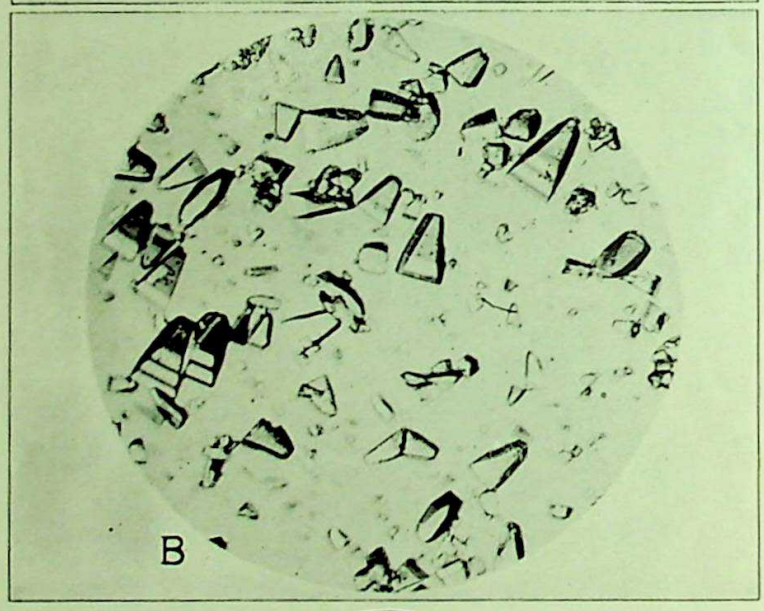
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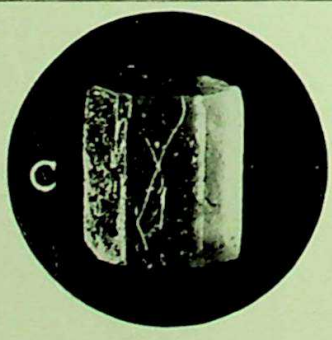
C

## Plate 138

A.—Photomicrograph of the isolated "sand" crystals from some of the same ice cream used for Plate 137. Photographed with polarized light. × 90.

B.—Photomicrograph of some pure recrystallized lactose hydrate from condensed whey. Photographed with polarized light. × 90.

C.—Photomicrograph of a crystal of pure sucrose. Photographed in plane light. × 5.

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